

TITLE OF THE INVENTION

**PROCESS FOR ELECTROSTATOGRAPHIC REPRODUCTION**

INVENTORS

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**PROCESS FOR ELECTROSTATOGRAPHIC REPRODUCTION**  
**CROSS-REFERENCE TO RELATED APPLICATIONS**

**[01]** This Application claims benefit under 35 USC §119(e) of prior co-pending U.S. Provisional Patent Application, Serial No. 60/425,626, filed November 13, 2002. This provisional patent application is incorporated herein in its entirety, by reference thereto.

**[02]** Filed concurrently with this application are the application entitled "Layer Comprising Nonfibrillatable and Autoadhesive Particles, and Method of Preparation", Applicants' Docket Nos. 10159 and PAT00005, and the application entitled "Fuser Member And Fuser Member Surface Layer", Applicants' Docket Nos. 10167 and PAT00010. These two concurrently filed applications are incorporated herein in their entireties, by reference thereto.

**BACKGROUND OF THE INVENTION**

**1. Field of the Invention**

**[03]** The present invention relates to electrostatographic imaging and recording apparatus, and to assemblies in these apparatus for fixing toner to the substrates. The present invention relates particularly to a fuser member, and to a fusing surface layer for fuser members, in the toner fixing assemblies.

**2. Description of Background and Other Information**

**[04]** Generally in electrostatographic reproduction, the original to be copied is rendered in the form of a latent electrostatic image on a photosensitive member. This latent image is made visible by the application of electrically charged toner.

**[05]** The toner thusly forming the image is transferred to a substrate - also referred to in the art as a receiver - such as paper or transparent film, and fixed or fused to the

substrate. Where heat softenable toners - for example, comprising thermoplastic polymeric binders - are employed, the usual method of fixing the toner to the substrate involves applying heat to the toner, once it is on the substrate surface, to soften it, and then allowing or causing the toner to cool. This application of heat in the fusing process is preferably at a temperature of about 90°C-220°C; pressure may be employed in conjunction with the heat.

**[06]** A system or assembly for providing the requisite heat and pressure customarily includes a fuser member and a support member. The heat energy employed in the fusing process generally is transmitted to toner on the substrate by the fuser member. Specifically, the fuser member is heated; to transfer heat energy to toner situated on a surface of the substrate, the fuser member contacts this toner, and correspondingly also can contact this surface of the substrate itself. The support member contacts an opposing surface of the substrate.

**[07]** Accordingly, the substrate can be situated or positioned between the fuser and support members, so that these members can act together on the substrate to provide the requisite pressure in the fusing process. In cooperating, preferably the fuser and support members define a nip, or contact arc, in which the substrate is positioned or resides, and/or through which the substrate passes. Also as a matter of preference, the fuser and support members are in the form of fuser and pressure rollers, respectively. Yet additionally as a matter of preference, one or both of the fuser and support members have a soft layer that increases the nip, to effect better transfer of heat to fuse the toner.

**[08]** In contacting toner on the substrate, the surface of the fuser member imparts a surface texture to the toner, and accordingly to the image formed thereby. This surface texture determines the degree of image gloss; differences in the texture of the toner results in varying gloss levels.

**[09]** With glossy images formed by the indicated fusing process, there are certain disadvantages. Subtle defects can be seen in these images, particularly defects that cause subtle variations in gloss.

**[010]** However, matte images, or low gloss images, minimize objectionable glare, and they reduce or even hide various image defects, such as oil defects. Particularly, low gloss images can make subtle defects invisible.

**[011]** Where the receiver is paper, low gloss images are further advantageous in that they do not produce objectionable differential gloss due to nonuniformity in the paper height. Varying types of paper have different levels of roughness, and glossy images produced on rough paper often produce differential gloss. This is particularly a problem in high speed digital printing, where high paper speeds require short fusing times. In the faster printers, when the image is high gloss there is often not time to allow a uniform gloss image on rough papers. As the final operation for fixing the image to the receiver, fusing usually produces the final image surface. Thus, altering the fusing process can be necessary to obtain a desirable image property, such as low image gloss.

**[012]** Heavily filled silicone rubber, used for fuser member surfaces, is known to produce high quality fused images with the desired low gloss. The polysiloxane elastomers have relatively low surface energies and also relatively low mechanical strengths, but are adequately flexible and elastic.

Unfortunately, silicone rubbers wear easily when employed for this purpose; after a period of use, the action of the paper or other media passing through a high pressure nip wears a polysiloxane elastomer fuser surface. The silicone rubbers' low wear resistance as fuser member surfaces accordingly limits fuser member life. Further, although treatment with a polysiloxane release fluid during use of the fuser member enhances its ability to release toner, the fluid causes the silicone rubber to swell. This fluid absorption is a particular factor that shortens fuser member life; fluid treated portions tend to swell and wear and degrade faster. Fuser members with polysiloxane elastomer fusing surfaces accordingly have a limited life.

**[013]** Fluorocarbon materials also have low surface energies, and, like silicone rubbers, are used as release surface materials for fuser members. Polyfluorocarbons employed for this purpose include nonelastomeric fluorocarbon materials, or fluoroplastics, and fluoroelastomer materials. However, there are disadvantages associated with the use of both.

**[014]** U.S. Patents Nos. 5,363,180, 5,508,138, 5,519,479, 5,649,273, these four patents being incorporated herein in their entireties, by reference thereto, disclose a fixing device which is for use in an electrophotographic apparatus, and which includes a fixing roller. The fixing roller can have a surface layer, with particles harder than the layer dispersed therein. This surface layer can comprise a heat resistant fluororesin such as ethylene tetrafluoride, or a fluorine type heat contracting resin such as tetrafluoroethylene-perfluoroalkylvinylether copolymer.

**[015]** In fact, the fluorocarbon resins like polytetrafluoroethylene (PTFE), and copolymers of tetrafluoroethylene (TFE) and perfluoroalkylvinylether (PFA), and fluorinated

ethylene propylene copolymers, have excellent release characteristics due to very low surface energies. They also are characterized by high temperature resistance, excellent chemical resistance, and low wear (high abrasion resistance).

**[016]** However, fluorocarbon resins are less flexible and elastic than polysiloxane elastomers, and are unsuitable for producing high image quality images. Fluorocarbon resins typically have a high modulus, and cannot evenly contact rough papers; they therefore provide varying gloss within the same image. The high modulus also tends to produce images with objectionable mottle, and contributes to high gloss; specifically, with both a smooth surface and high modulus, there will be high gloss in addition to the objectionable mottle.

**[017]** Fluoroelastomers also have low surface energy. They have excellent wear resistance as fusing member surfaces, providing better durability in this regard than the polysiloxane elastomers, and unlike the silicone rubbers, do not swell when in contact with polysiloxane release fluids. However, due to their relatively greater hardness as compared with that of the silicone rubbers, fluoroelastomers also typically produce objectionably high gloss images.

**[018]** It would accordingly be desirable to provide a fusing member fluoroelastomer surface which retains the indicated advantages of fluoroelastomers, while also producing low gloss and high quality images as are achieved with silicone rubbers. It would further be desirable that obtaining the requisite low gloss not depend on the properties of the substrate, but rather be accomplished over a range of substrate surface roughness.

**[019]** Moreover, it is known that the presence of heat conducting inorganic filler particles, in the fluoroelastomer fusing surface layers of fuser members, provides high energy sites for removing toner from the substrate. Increasing the amount of heat conducting filler content in the fusing surface layer, by providing more reactive sites for the toner, therefore increases toner offset, and accordingly also increases contamination of the fuser member. Thus it would yet additionally be desirable that, where heat conducting inorganic filler particles are present in the fluoroelastomer fusing surface layer, the amount of the filler be minimized.

#### SUMMARY OF THE INVENTION

**[020]** It has been discovered that a fluoroelastomer fusing surface layer with a relatively smooth surface, and without the necessity of incorporating particles that attract toner, can be used for generating low gloss images. The fusing surface layer fulfilling these objectives comprises domains, particularly soft domains, within the fluoroelastomer.

**[021]** The present invention allows for the use of wear resistant and low surface energy fluoroelastomer, in generating highly desirable low gloss images that do not produce glare. Image defects, such as release oil artifacts and differential wear from skives or sensors contacting the roller surface, are not visible.

**[022]** The benefits as indicated are not dependent upon the substrate surface to which the toner is fused. Particularly, they are obtained with paper regardless of its roughness, or whether it is coated or uncoated; at least, low gloss is provided over a range of substrate roughness. Still further, generation of low gloss is maintained despite wear on the fuser member surface. And even on rough substrates, the gloss

obtained is uniform, or at least essentially or substantially uniform.

**[023]** The invention pertains to a process for fusing toner residing on a substrate to the substrate. The process comprises transmitting heat to the toner, and also contacting the toner with the fusing surface layer of a fuser member.

**[024]** The fuser member comprises a base, and a fusing surface layer. The fusing surface layer comprises a fluoroelastomer continuous phase, and a discontinuous phase dispersed through the fluoroelastomer continuous phase in the form of domains.

**[025]** At the temperature of the fusing process, the modulus of the fluoroelastomer continuous phase is greater than the modulus of the discontinuous phase. Also, the discontinuous phase comprises at least a minimum proportion by volume of the fusing surface layer and/or at least a minimum proportion by weight of the fluoroelastomer continuous phase, and, at the fusing process temperature, the difference between the modulus of the fluoroelastomer continuous phase and the modulus of the discontinuous phase is sufficiently great, the viscosity of the toner is sufficiently high, and the modulus of the discontinuous phase is sufficiently low, so that the image generated in the process has a gloss number of about 10 or less - more preferably of about 8 or less, still more preferably of about 6 or less, and still more preferably of about 5 or less. Yet additionally, in the process the amount of heat transmitted to the toner is sufficient to fuse the toner to the substrate, and insufficient to raise the gloss number of the image above about 10 or less - correspondingly, more preferably above about 8 or less, still more preferably above about 6 or less, and still more preferably above about 5 or less.



**[026]** Preferably, the process further comprises heating the fuser member, with the transmission of heat to the toner comprising the contacting of the toner with the fusing surface layer. Also as a matter of preference, the fuser member cooperates with a support member - both to define a nip in which the toner residing on the substrate is positioned the fuser member, and also to exert pressure, in the nip, on the substrate and on the toner residing thereon - with the contacting of the toner by the fusing surface layer occurring in the nip.

#### BRIEF DESCRIPTION OF THE DRAWINGS

**[027]** Fig. 1 is a schematic representation, and a sectional view, of a toner fusing assembly of the invention.

**[028]** Fig. 2 is a schematic representation, and an enlarged fragmentary sectional view, of an embodiment of the fuser member of the invention.

**[029]** Fig. 3 is a schematic representation, and an enlarged fragmentary sectional view, of another embodiment of the fuser member of the invention.

#### DESCRIPTION OF THE INVENTION

**[030]** Copolymers are understood as including polymers incorporating two monomeric units, i.e., bipolymers, as well as polymers incorporating three or more different monomeric units, e.g., terpolymers, quaterpolymers, etc.

**[031]** Polyorganosiloxanes are understood as including functional and nonfunctional polyorganosiloxanes. Polyorganosiloxanes further are understood as including polydiorganosiloxanes - i.e., having two organo groups attached to each, or substantially each, or essentially each, of the polymer siloxy repeat units. Polyorganosiloxanes yet further are understood as including polydimethylsiloxanes.

**[032]** Functional polyorganosiloxanes are understood as being polyorganosiloxanes having functional groups on the backbone, connected to the polysiloxane portion, which can react with fillers present on the surface of the fuser member, or with a polymeric fuser member surface layer or component thereof. Functional polyorganosiloxanes further are understood as being polyorganosiloxanes having functional groups such as amino, hydride, halo (including chloro, bromo, fluoro, and iodo), carboxy, hydroxy, epoxy, isocyanate, thioether, and mercapto functional groups. Nonfunctional polyorganosiloxanes further are understood as being polyorganosiloxanes without groups of the type as indicated.

**[033]** The term "organo" as used herein, such as in the context of polyorganosiloxanes, includes "hydrocarbyl", which includes "aliphatic", "cycloaliphatic", and "aromatic". The hydrocarbyl groups are understood as including the alkyl, alkenyl, alkynyl, cycloalkyl, aryl, aralkyl, and alkaryl groups. Further, "hydrocarbyl" is understood as including both nonsubstituted hydrocarbyl groups, and substituted hydrocarbyl groups, with the latter referring to the hydrocarbyl portion bearing additional substituents, besides the carbon and hydrogen. Preferred organo groups for the polyorganosiloxanes are the alkyl, aryl, and aralkyl groups. Particularly preferred alkyl, aryl, and aralkyl groups are the C<sub>1</sub>-C<sub>18</sub> alkyl, aryl, and aralkyl groups, particularly the methyl and phenyl groups.

**[034]** Gloss number is defined as the value measured using a BYK-Gardner (GB-4520 micro-tri-gloss) meter at an angle of 85 degrees from the vertical on a solid toned area. The gloss meter reading is the percentage of white light reflected from a test sample relative to a black glass

standard with a refractive index of about 1.567 (this measurement conforms to ASTM D 523 Standard Test Method for Specular Gloss). At 85 degrees, about 99 percent of incident light is reflected by the black glass standard so the gloss number is approximately equal to the percentage of reflected light. A solid toned area is defined as having a reflection density equal to or greater than 1.0 using an X-Rite 404 Reflection Densitometer, from X-Rite Company, Grand Rapids, MI. For conducting the indicated measurements, 20# bond paper is employed; however, with these procedures, similar results would still be obtained using smoother papers.

**[035]** Fusing or operating temperatures, or the temperature of the fusing process, are understood as being within the range of from about 90°C, or about 120°C, or about 150°C, to about 200°C, or about 220°C, or about 250°C. The preferred temperatures are generally within the range of from about 120°C to about 200°C, more preferably from about 150°C to about 175°C, still more preferably from about 150°C to about 185°C.

**[036]** Unless stated otherwise, modulus is measured as tensile modulus of elasticity, using dynamic mechanical analysis, at a frequency equal to the frequency of the fuser member. The fuser member frequency pertains to the 360° rotation of the member - in the case of fuser members that operate in the fusing process by rotation (e.g., fuser members with bases in the form of cylindrical cores, or of belts on rollers, or of core-mounted plates) - and in the case of other fuser members, to their equivalent movement.

**[037]** Equilibrium surface roughness, of the fusing surface layer, is the surface roughness where the degree of roughness remains unchanged, or essentially unchanged, as use of the fuser member in the fusing process proceeds. At equilibrium surface roughness, wearing away of the fusing surface layer at

its surface regenerates a surface with the same, or essentially the same, degree of roughness.

**[038]** The fuser member includes a fuser base, and a fusing surface layer overlaying the fuser base. The fusing surface layer can reside directly on the fuser base. Alternatively, there can be one or more materials and/or layers, including one or more cushion layers, interposed between the fuser base and the fusing surface layer.

**[039]** The fusing surface layer comprises a fluoroelastomer continuous phase, and a discontinuous phase dispersed through the fluoroelastomer continuous phase in the form of domains. The modulus of the discontinuous phase, and particularly of the domains comprising this phase, is lower than the modulus of the fluoroelastomer continuous phase, particularly at fuser operating conditions, or at the temperature of the fusing process, or at the fusing temperature.

**[040]** Also particularly at fuser operating conditions, or at the temperature of the fusing process, or at the fusing temperature, the fusing surface layer preferably has a modulus of about  $2 \times 10^7$  Pa or less, or about  $1 \times 10^7$  Pa or less - or from about  $1 \times 10^6$  Pa, or about  $5 \times 10^6$  Pa, to about  $1 \times 10^7$  Pa, or about  $2 \times 10^7$  Pa. However, in particular embodiments, this layer may have a modulus of up to about  $1 \times 10^8$  Pa, or up to about 2 or about  $3 \times 10^8$  Pa, or higher.

**[041]** Again, at fuser operating conditions, or at the temperature of the fusing process, or at the fusing temperature, the fluoroelastomer continuous phase preferably has a modulus of about  $5 \times 10^8$  Pa or less, or about  $2 \times 10^8$  Pa or less - or from about  $8 \times 10^6$  Pa to about  $2 \times 10^8$  Pa, or about  $5 \times 10^8$  Pa.

**[042]** As a matter of preference, the domains of the discontinuous phase, at the conditions - particularly

temperature - as indicated, are in the form of solids, with the discontinuous phase accordingly preferably being a solid. As a matter of particular preference, at the indicated conditions the discontinuous phase is in the form of soft domains, and particularly in the form of soft solid domains.

**[043]** Preferably the discontinuous phase comprises at least one polymer. Preferred polymers for the discontinuous phase are the elastomers.

**[044]** Among the elastomers that may be used are perfluoropolyethers, fluoroelastomers, and particularly silicones, such as fluorosilicones. Suitable silicone elastomers include those that may be used for the optional one or more cushion layers, as discussed.

**[045]** Suitable fluoroelastomers include those that may be used for the continuous phase of the fusing surface layer, as discussed. Where the discontinuous phase comprises a fluoroelastomer, the fluoroelastomers of the continuous and discontinuous phases must be such that, at the temperature of the fusing process, the modulus of the fluoroelastomer continuous phase is greater than the discontinuous phase modulus, and the difference between the modulus of the fluoroelastomer continuous phase and the modulus of the discontinuous phase is sufficiently great, and the modulus of the discontinuous phase is sufficiently low, so that the requisite gloss level is achieved, as discussed.

**[046]** The discontinuous phase material can be provided, for use in preparing the fusing surface layer, already in final form, and in this final form, serve as the domains dispersed in fluoroelastomer. Particularly, the discontinuous phase material may be provided already in particulate form.

**[047]** In this regard, the elastomers as indicated may be provided as preformed particles. Particularly, preformed

silicone elastomer particulate may be used. The silicone elastomer particles are crosslinked particles of polydiorganosilixane, preferably polydimethylsiloxane, elastomer.

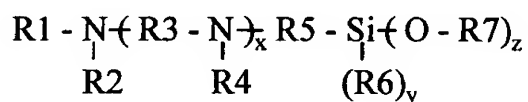
**[048]** The silicone elastomer particulate can be prepared by emulsion polymerization, or from bulk silicone by grinding, or otherwise reducing the material to discrete particles. Particularly, the crosslinked polydiorganosiloxane - e.g., polydimethylsiloxane - particles can be obtained using condensation or addition cure methods. Preparation preferably involves the reaction of a vinyl dimethyl terminated polydimethylsiloxane having a number average molecular weight of from about 2,000 to about 20,000, and either a polymethylhydrosiloxane or a methyl hydro, dimethylsiloxane copolymer, having a number average molecular weight of from about 300 to about 3,000; also as a matter of preference, the ratio of hydride to vinyl groups is from about 1:1 to about 2:1.

**[049]** The silicone elastomer particles particularly can be prepared in accordance with the procedures set forth in U.S. Patent No. 6,281,279; accordingly, the silicone elastomer particles as disclosed in U.S. Patent No. 6,281,279 can be used. This patent is incorporated herein in its entirety, by reference thereto.

**[050]** In a preferred embodiment, the silicone elastomer particles are in a core-shell configuration, comprising a silicone resin shell surrounding the silicone elastomer core. The shell is expected to provide the particle with a surface of higher hardness to the particle, which will also have a higher surface energy. This surface treated preformed silicone particulate may have better adhesion with the

surrounding elastomer matrix, while retaining the advantage of added silicone rubber.

**[051]** The surface treatment, to provide the shell portion of the core-shell configuration, can be performed *in situ* by adding an aminosilane to the coating preparation. The amount of aminosilane added is from about 0.1 percent by weight to about 5 percent by weight of the silicone particles. Preferably the aminosilane has the formula



wherein

$\text{R}^1$  and  $\text{R}^2$  are the same or different, and are selected from the group consisting of hydrogen and  $\text{C}_1\text{-C}_8$  hydrocarbyl groups;

$\text{R}^3$  is a  $\text{C}_1\text{-C}_8$  hydrocarbyl group;

$\text{R}^4$  is selected from the group consisting of hydrogen and  $\text{C}_1\text{-C}_6$  hydrocarbyl groups;

$\text{R}^5$  is a  $\text{C}_3\text{-C}_8$  hydrocarbyl group;

$\text{R}^6$  and  $\text{R}^7$  are the same or different, and are selected from the group consisting of  $\text{C}_1\text{-C}_8$  hydrocarbyl groups;

$x$  is 0 to 2;

$y$  is 0 to 2;

$z$  is 1 to 3; and

$y + z$  is 3.

**[052]** Commercially available silicone particulates that are suitable for the invention include 52-854, X-52-875, KMP597, and KMP598, from Shin-Etsu Silicones of America, Inc., Akron, OH. A commercially available core-shell configuration silicone particulate that is suitable is KMP600, also from Shin-Etsu Silicones.

**[053]** The material - e.g., the one or more polymers - that becomes the discontinuous phase, particularly elastomers used for this purpose, may be employed in cured form, or in curable form, such as a curable liquid or gum. Where employed in curable form, the material is cured by the curing of the fusing surface layer of the invention, to provide the discontinuous phase domains; particularly, the curing of the layer effects crosslinking of curable polymer that is present.

**[054]** Among the curable silicone elastomers that may be employed are peroxide cure silicone elastomers and vinyl addition cure silicone elastomers. Particularly preferred for the discontinuous phase are the curable siloxane polymers, particularly the curable polyfunctional poly(C<sub>1-6</sub> alkyl) siloxane polymers, disclosed in U.S. Patent No. 5,582,917, which is incorporated herein in its entirety, by reference thereto.

**[055]** A preferred commercially available curable siloxane polymer is SFR-100 silicone, from GE Silicones, Waterford, NY. SFR-100 silicone is characterized as a silanol- or trimethylsilyl-terminated polymethylsiloxane, and is a liquid blend comprising about 60-80 weight percent of a difunctional polydimethylsiloxane having a number average molecular weight of about 150,000, and 20-40 weight percent of a polytrimethylsilyl silicate resin having monofunctional (i.e. trimethylsiloxane) and tetrafunctional (i.e. SiO<sub>2</sub>) repeating units in an average ratio of between about 0.8 and 1 to 1, and having a number average molecular weight of about 2,200.

**[056]** The curable elastomers, and particularly the curable siloxane polymers, can be employed in the amount of up to about 50 parts, or up to about 60 parts, or up to about 80 parts, per 100 parts by weight of the fluoroelastomer. Particularly, the curable elastomers, and more particularly



the curable siloxane polymers, may be used in the amount of from about 5 parts, or about 10 parts, to about 50 parts, or about 60 parts, or about 80 parts, or about 100 parts, per 100 parts by weight of the fluoroelastomer.

**[057]** At fuser operating conditions, or at the temperature of the fusing process, or at the fusing temperature, the domains of the discontinuous phase preferably have a modulus of about  $8 \times 10^6$  Pa or less, or about  $10 \times 10^6$  Pa or less, or from about  $0.5 \times 10^6$  Pa, or about  $0.8 \times 10^6$  Pa, or about  $1 \times 10^6$  Pa, to about  $4 \times 10^6$  Pa, or about  $8 \times 10^6$  Pa, or about  $10 \times 10^6$  Pa. The discontinuous phase domains preferably have a mean diameter - particularly in the case of the particulate, a mean particle diameter - of from about 0.1 microns, or about 0.2 microns, or about 0.5 microns, or about 1 micron, to about 30 microns, or about 40 microns, or about 50 microns, or about 60 microns, or about 80 microns. The domains of the discontinuous phase preferably are present, in the fusing surface layer, in an amount of from about 10 parts to about 80 parts, or about 100 parts, or about 120 parts, or about 150 parts, per 100 parts by weight of the fluoroelastomer continuous phase.

**[058]** The material for forming the discontinuous phase - provided already in final form, or provided in curable form, to be cured to form the domains - can be included with the materials that are compounded. However, preferably the material for forming the discontinuous phase is added to the solvent with which the compounded materials are subsequently combined, as discussed herein.

**[059]** Fillers typically are characterized by high surface energy and by a high degree of hardness; inorganic particles in particular customarily have these properties. As discussed, toner tends to adhere to high surface energy

particles and fillers, and they tend to abrade toner fusing system elements provided to contact the fuser member. Further, where filler is absent or at sufficiently low levels in the fusing surface layer, and this layer also has a modulus sufficiently low, even contacting of the toner by the fusing surface layer is facilitated, despite high variations in the surface of the substrate on which the toner resides. Accordingly, in general it is desirable to minimize the amount of filler - particularly filler having these properties, and particularly inorganic filler and plastic filler - in the fusing surface layer, or even to provide a fusing surface layer from which this filler is absent, or essentially absent.

**[060]** Nevertheless, notwithstanding the foregoing, the fusing surface layer further may include one or more fillers, for one or more purposes. Different fillers may be used for such purposes as conducting heat, improving toner offset and release properties of the fusing surface layer, controlling material properties such as wear resistance and surface roughness, modifying hardness, and imparting other characteristics, such as desired mechanical properties, to the fusing surface layer; among the fillers which may be included are reinforcing fillers.

**[061]** Fillers that are suitable include inorganic fillers, such as  $\text{SnO}_2$ ,  $\text{SiC}$ ,  $\text{CuO}$ ,  $\text{ZnO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{FeO}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{WC}$ ,  $\text{BN}$ , and amorphous silica, such as precipitated silica and fumed silica. Further fillers that are suitable include plastic fillers. The plastics are understood as including non-crosslinked plastics, and also as including resins, particularly inelastic resins, and crosslinked resins. Fluoroplastics are particularly preferred, as are fluororesins, or nonelastomeric fluorocarbons. Fluororesins that are suitable include polytetrafluoroethylenes (PTFE), and

fluorinated ethylene propylenes (FEP), including copolymers of tetrafluoroethylene and hexafluoropropylene, as well as copolymers of tetrafluoroethylene and ethylene, and copolymers of tetrafluoroethylene and perfluoroalkyl vinyl ether (PFA). Preferably the plastics, and particularly the fluororesins, have a number average molecular weight of from about 1,000 to 1,000,000.

**[062]** These one or more fillers may be present in amounts and proportions, and sizes, as are generally known or as can be determined without undue experimentation by those of ordinary skill in the art. Where present, filler as indicated preferably comprises not more than about 35 percent by volume, more preferably not more than about 25 percent by volume, of the fusing surface layer. Still more preferably, filler as indicated comprises from about 5 percent by volume to about 25 percent by volume, or to about 35 percent by volume, of the fusing surface layer.

**[063]** Where filler is present, as a matter of particular preference, the filler comprises not more than about 35 percent by volume of the fusing surface layer, and also the fusing surface layer has a modulus of about  $2 \times 10^7$  Pa or less at fuser operating conditions, or at the temperature of the fusing process, or at the fusing temperature. Still more preferably where filler is present, the filler comprises not more than about 25 percent by volume of the fusing surface layer, and also the fusing surface layer has a modulus of about  $1 \times 10^7$  Pa or less at fuser operating conditions, or at the temperature of the fusing process, or at the fusing temperature.

**[064]** The one or more fillers may be in one or more of any suitable shapes - irregular, as well as in the form of spheroids, platelets, flakes, powders, ovoids, needles,

fibers, and the like. For filler which is employed for the purpose of providing thermal conductivity, and particularly in such instance and where internal heating is employed, an irregular shape is more preferred, as are spherical particles and platelets, so as to maximize the heat conducting effect of the filler particles; fibers, needles, and otherwise elongated shapes are less preferred here, unless they are advantageously oriented, because in certain alignments they are less effective for properly conducting heat.

**[065]** In this regard, elongated particles are more efficient for conducting heat in the proper direction if they are at right angles to the fuser base - radially aligned, if the fuser base is a cylindrical core, belt on rollers, or a core-mounted plate, but less efficient if they are positioned parallel to the core - axially aligned, if the fuser base is a core, a belt, or is core mounted as indicated. Accordingly, to maximize heat conducting properties where elongated heat conducting particles are employed, perpendicular (radial) positioning is preferred, while parallel (axial) alignment may be employed but is not preferred.

**[066]** Particularly as to sizes, preferably these one or more fillers have a mean particle diameter of from about 0.1 microns to about 80 microns, more preferably of from about 0.2 microns to about 50 microns. Particularly, the indenter particles as disclosed in the application identified herein as Applicants' Docket Nos. 10167 and PAT00010, can be included in the fusing surface layer. For instance, the indenter particles may be employed in the amounts and/or proportions, and sizes, as disclosed in the application

identified herein as Applicants' Docket Nos. 10167 and PAT00010.

**[067]** Filler that is employed - e.g., inorganic filler and plastic filler, and particularly indenter particles, including inorganic indenter particles and plastic indenter particles - preferably is insoluble in the solvents used for preparing the coating preparations of the invention. Discontinuous phase material that, when employed, is already in final form - e.g., preformed particulate, and particularly silicone elastomer particles - likewise is preferably insoluble. In this regard, these materials preferably are insoluble even to the extent that the solvents cannot make the particles adhere to one another due to softening.

**[068]** Yet additionally, the indicated filler and final form discontinuous phase material preferably are heat stable at fusing or operating temperatures. Particularly, these materials preferably are heat stable at fusing process temperatures - e.g., within the range of from about 90°C, or about 120°C, or about 150°C, to about 200°C, or about 220°C, or about 250°C. Accordingly, the discontinuous phase material and filler preferably are heat stable at temperatures of up to at least about 90°C, more preferably up to at least about 120°C, still more preferably up to at least about 150°C, still more preferably up to at least about 200°C, still more preferably up to at least about 220°C, or even up to at least about 250°C.

**[069]** This heat stability entails the absence, or at least the essential absence or substantial absence, of degradation, decomposition, sublimation, and release of byproducts, and of change in shape, size, or state of matter. And indenter particles, where employed, in addition to being thusly heat stable, also undergo no melting, or

essentially or substantially no melting, at the temperatures as indicated.

**[070]** For improving the wear resistance and release properties of the fusing surface layer, one or more of the materials which are used for preparing the fusing surface layer, and which are reactive with SiOH groups, may be compounded with a coupling agent - preferably a silane coupling agent, as discussed in U.S. Patent No. 5,998,033. Materials suitable for this treatment include inorganic fillers and cocuratives.

**[071]** As to this matter, herein it is disclosed that the materials which are compounded, for subsequent combination with solvent and formation of the fusing surface layer, include the fluoroelastomer for preparing the continuous phase. Where filler and cocurative are being employed, they also may be included in the dry compounding treatment. Accordingly, where one or more SiOH group-reactive materials, as indicated, are present, the requisite amount of coupling agent yet additionally can be included in the compounding of these materials.

**[072]** Instead of compounding with coupling agent, one or more of the SiOH group-reactive materials may be surface treated with a coupling agent - here also preferably a silane coupling agent, as discussed in U.S. Patents Nos. 5,935,712, 6,090,491, and 6,114,041. The coupling agent can be dissolved in an appropriate solvent, and surface treatment can be effected by steeping the material in this solution; ultrasonication can be employed during this treatment. After treatment the material is washed and dried. In the case of silane, preferably the treatment solution is prepared by adding about 2 weight percent of this coupling agent to a solvent comprising 95 percent by volume ethanol and 5 percent

by volume water, and stirring for ten minutes. The material is covered by the solution and ultrasonicated for ten minutes. The material then is separated by vacuum filtration, rinsed with ethanol, and thereafter oven dried at 150°C, for 18 hours under reduced pressure (vacuum).

**[073]** It is understood that both the surface treatment and the compounding, as discussed, are included in referring to treatment with coupling agent. It is further understood that both material compounded with silane coupling agent, and material surface treated with silane coupling agent, are included in referring to the resulting product as silane coupling agent-treated material.

**[074]** Particularly as to the silane coupling agents, 3-aminopropyltriethoxysilane is a silane which may be employed. However, the secondary amine functional silanes are preferred, because they have relatively less of an unfavorable impact upon pot life. Suitable secondary amine functional silanes include N-phenylaminopropyltrimethoxysilane, N-phenylaminopropyltriethoxysilane, 3-[2-N-benzylaminoethylaminopropyltrimethoxysilane, and 3-[2-N-benzylaminoethylaminopropyltriethoxysilane. Also among the silanes which may be used are the styryl-functionalized silane coupling agents disclosed in U.S. Patent No. 6,090,491.

**[075]** U.S. Patents Nos. 5,998,033, 5,935,712, 6,090,491, and 6,114,041 are incorporated herein in their entireties, by reference thereto.

**[076]** Yet other additives and adjuvants also may be used, as long as they do not affect the integrity of the fluoroelastomer for the continuous phase, or significantly interfere with an activity intended to occur in the fusing surface layer - such as the crosslinking of the fluoroelastomer. These further additives and adjuvants, where

present, are provided in amounts and proportions as are generally known or as can be determined without undue experimentation by those of ordinary skill in the art. Suitable examples include crosslinking agents, processing aids, accelerators, polymerization initiators, and coloring agents.

**[077]** The fuser base may be any of those as are known in the art. As a suitable embodiment, the fuser base may be a core in the form of a cylinder or a cylindrical roller, particularly a hollow cylindrical roller. In this embodiment the fuser base may be made of any suitable metal, such as aluminum, anodized aluminum, steel, nickel, copper, and the like. Also appropriate are ceramic materials and polymeric materials, such as rigid thermoplastics, and thermoset resins with or without fiber enforcement. Preferably the roller is an aluminum tube or a flame sprayed aluminum coated steel tube.

**[078]** Alternatively, the fuser base may be a plate. Materials suitable for the core may also be used for the plate.

**[079]** One embodiment of a fuser base in the plate form is a curved plate mounted on a larger cylindrical roller - that is, larger than a cylindrical roller which itself is employed as a fuser core. Being thusly curved, the plate accordingly has the shape of a portion of a cylinder. Additionally, the plate can be removably mounted on the cylindrical roller, so that the plate can be replaced without also requiring replacement of the roller. In this embodiment, the properties discussed herein with reference to the fuser base pertain only to the portion of the cylindrical roller occupied by the attached plate; the rest



of this roller is not involved in the fusing of toner to substrate.

**[080]** As yet another alternative, the fuser base may be a belt, particularly an endless flexible belt. A thin belt made of a suitable metal, such as those indicated for the core and plate forms; the belt may also be made of a polyamide or a polyimide, particularly a heat resistant polyamide or polyimide. A polyimide material appropriate for the belt is commercially available under the trademark Kapton, from DuPont High Performance Films, Circleville, OH.

**[081]** Preferably the belt is mounted on rollers, which can be cores of the type as discussed herein. As a matter of preference two rollers are utilized with the belt, each of these two rollers defining a different one of the curves around which the belt passes.

**[082]** A support member for the fusing system and process likewise may be any of those as are known in the art; particularly, it can be a backup roller, also referred to as a pressure roller. The support member can be in the form of a roller, plate, or belt, in the same manner as is suitable for the fuser base; particularly, cores suitable for the fuser member may also be used for the support member. Where the support member is a belt, preferably it is mounted on rollers, in the same manner as - for the fuser base in the form of a belt.

**[083]** In any of the indicated forms, the support member may have mounted thereon a cushion for forming the nip with the fuser member. Suitable cushion materials include those having at least some degree of temperature resistance, such as silicone and EPDM elastomers. In the absence of yet a further layer in turn being mounted on the cushion, this

cushion also serves to contact the substrate, and accordingly to cooperate with the fuser member.

**[084]** Alternatively or in addition to the cushion, the support member may have mounted thereon a thin fluoroplastic surface layer, such as a Teflon or PFA layer, overlaying the surface that cooperates with the fuser member. Where both the cushion and the thin fluoroplastic surface layer are present on the support member, the cushion is situated between the support member and the surface layer.

**[085]** Preferably the fuser base is in the form of a cylindrical roller, with the fuser member correspondingly in the form of a roller - specifically, a fuser roller. Also as a matter of preference, the support member comprises a backup roller.

**[086]** Internal heating and/or external heating may be employed in the toner fusing system and process. Heating means as are known in the art are suitable. Preferably, the means of providing heat for fusing toner and substrate comprise the heating of the fuser member by one or more external and/or internal heating sources, and transmission of this heat from the fuser member to the toner, or to both toner and substrate - preferably by contact.

**[087]** Where there are one or more materials and/or layers, including one or more cushion layers, interposed between the fuser base and the fusing surface layer, they may be those as are known in the art. Where there is at least one cushion layer, the at least one cushion layer can include one or more thermally conductive cushion layers and/or one or more thermally nonconductive cushion layers.

**[088]** When used, the at least one cushion layer particularly can be that as disclosed in U.S. Application No. 09/879,585, filed June 12, 2001; this application is incorporated herein

in its entirety, by reference thereto. Generally, the thickness of the at least one cushion layer is about 20 millimeters or less, preferably from about 1 to about 10 millimeters.

**[089]** Among the materials which can be used for the at least one cushion layer are suitable silicone elastomers, such as appropriate thermally conductive silicone elastomers and thermally nonconductive silicone elastomers. Addition cure, condensation cure, and peroxide cure silicone elastomers can all be used, with addition cure silicone elastomers and condensation cure silicone elastomers being preferred.

**[090]** Further, silicone elastomers formulated as room temperature vulcanizate (RTV), liquid injection moldable (LIM), and high temperature vulcanizate (HTV) silicone elastomers can be used. RTV and LIM silicones are preferred.

**[091]** A highly desired property for the silicone elastomers is heat stability. Particularly for cushion layer silicone elastomers, this property is characterized by low compression set, resistance to hardening or softening over time, and resistance to tear propagation from heat aging.

**[092]** In particular, compression set is permanent deformation. Low compression set, or good compression set resistance, is required for the desired shape of the fuser roller to be maintained.

**[093]** Two particular silicone elastomers which may be used are Silastic™-J silicone, from Dow Corning Corporation, Midland, MI, and a silicone commercially available under the designation EC4952 from Emerson & Cuming ICI, Billerica, MA.

**[094]** In a process which may be used for application of at least one cushion layer, the fuser base optionally can first

be degreased and surface roughened. If these functions are performed, they may be accomplished by grit blasting. Except as discussed otherwise herein, the fuser base surface, whether or not initially degreased and roughened, is primed with conventional primer, such as Dow™ 1200 RTV Prime Coat primer, from Dow Corning Corporation, and material for forming a cushion is subsequently applied thereto.

**[095]** To form a cushion layer, silicone elastomer is molded, particularly by injection, or extruded or cast onto the fuser base to the desired thickness. Curing is then effected. For a RTV silicone, this is accomplished by allowing it to sit at room temperature.

**[096]** After curing, conventionally the silicone layer is subjected to a post cure, which improves compression set resistance. Typically a post cure is conducted at a temperature of around 200°C, or about 150-200°C, or about 200-230°C, or as high as about 240°C, for a period of about 1-2 hours, or for about 4 hours, or for about 24 hours, or for a period of about 4-48 hours.

**[097]** Each silicone cushion layer is subjected to cure, and preferably also to post cure, before application of the next layer, except in the case of the last silicone layer to be laid down. For this finally applied silicone cushion layer, the composition for forming the fusing surface layer is first laid down and then cured at a raised temperature for a period of time, as discussed herein.

**[098]** This curing serves as the post cure for the silicone cushion layer on which the indicated fusing surface layer composition is deposited. Delaying the post cure of the last cushion layer in this manner allows maximum adhesion between the cushion and the fusing surface layer to develop.

Where only one silicone cushion layer is employed, since it is also the last cushion layer to be laid down, it is not post cured until the fusing surface layer is applied, in accordance with the foregoing.

**[0099]** Before the composition for forming the fusing surface layer is applied, the cushion material can be ground to a desired profile, depending upon the paper handling concerns to be addressed. For instance, a cylinder shape, or a crown, or barrel, or bow tie, or hourglass profile may be provided.

**[0100]** Addition cure silicone elastomers typically employ a platinum catalyst; condensation cure silicone elastomers, a tin catalyst. Tin catalysts will poison platinum catalysts, but the reverse is not true. Accordingly, where sequential addition and condensation cure silicone elastomer layers are employed, a condensation cure layer can be applied onto an addition cure layer, but not vice versa.

**[0101]** Suitable fluoroelastomers for the fusing surface layer, and particularly for the continuous phase of the fusing surface layer, include random polymers comprising two or more monomeric units, with these monomeric units comprising members selected from a group consisting of vinylidene fluoride  $[-(\text{CH}_2\text{CF}_2)-]$ , hexafluoropropylene  $[-(\text{CF}_2\text{CF}(\text{CF}_3))-]$ , tetrafluoroethylene  $[-(\text{CF}_2\text{CF}_2)-]$ , perfluoro-vinylmethyl ether  $[-(\text{CF}_2\text{CF}(\text{OCF}_3))-]$ , and ethylene  $[-(\text{CH}_2\text{CH}_2)-]$ . Among the fluoroelastomers that may be used are fluoroelastomer copolymers comprising vinylidene fluoride and hexafluoropropylene, and terpolymers as well as tetra- and higher polymers including vinylidene fluoride, hexafluoropropylene, and tetrafluoroethylene monomeric units. Additional suitable monomers include perfluorovinyl-alkyl ethers, such as perfluorovinylmethyl ether.

**[0102]** Preferred fluoroelastomers include random polymers comprising the following monomeric units:

$-(\text{CH}_2\text{CF}_2)_x-$ ,  $-(\text{CF}_2\text{CF}(\text{CF}_3))_y-$ , and  $-(\text{CF}_2\text{CF}_2)_z-$ ,  
wherein  $x$  is from about 30 to about 90 mole percent,  
 $y$  is from about 10 to about 60 mole percent, and  
 $z$  is from about 0 to about 42 mole percent.

**[0103]** Further preferred fluoroelastomers are random polymers comprising the following monomeric units:

$-(\text{CH}_2\text{CH}_2)_x-$ ,  $-(\text{CF}_2\text{CF}(\text{OCF}_3))_y-$ , and  $-(\text{CF}_2\text{CF}_2)_z-$ ,  
wherein  $x$  is from about 0 to about 70 mole percent,  
 $y$  is from about 10 to about 60 mole percent, and  
 $z$  is from about 30 to about 90 mole percent

**[0104]** The fluoroelastomers, as discussed, may further include one or more cure site monomers. Among the suitable cure site monomers are 4-bromoperfluorobutene-1, 1,1-dihydro-4-bromo-perfluorobutene-1, 3-bromoperfluorobutene-1, and 1,1-dihydro-3-bromoperfluoropropene-1. When present, cure site monomers are generally in very small molar proportions. Preferably, the amount of cure site monomer will not exceed about 5 mole percent of the polymer.

**[0105]** The fluoroelastomer molecular weight is largely a matter of convenience, and is not critical to the invention. However, as a matter of preference, the fluoroelastomers have a number average molecular weight of from about 10,000 to about 200,000. More preferably they have a number average molecular weight of from about 50,000 to about 100,000.

**[0106]** Among the fluoroelastomers that may be used are those that are plastic at ambient temperature and elastomeric at fusing or operating temperatures.

**[0107]** Commercially available fluoroelastomers which may be used are those sold under the trademark Viton® by Dupont Dow Elastomers, Stow, OH; they include Viton® A, Viton® B, Viton®

E, Viton® GF, Viton® GH, Viton® GFLT, Viton® B 50, Viton® B 910, Viton® E 45, Viton® E 60C, and Viton® E 430. Also suitable are the Tecnoflons®, such as T838K, FOR-THF, FOR-TFS, FOR-LHF, NM, FOR-60KIR, TH, TH505, and FOR4391, from Ausimont USA, Inc., Thorofare, NJ, and the Fluorel™ fluoroelastomers, such as FE5840Q, FX9038, FX2530, FLS5840Q, FLS2690, FC2230, FC2145, FT2430, Fluroel 2170, Fluorel 2174, Fluorel 2177, Aflas (a polypropylene-tertafluoroethylene), and Fluorel II L11900 (a polypropylene-tetrafluoroethylene vinylidene fluoride), from Dyneon L.L.C., Oakdale, MN.

**[0108]** Appropriate fluoroelastomers include those as identified in U.S. Patents Nos. 4,372,246, 5,017,432, 5,217,837, and 5,332,641. These four patents are incorporated herein in their entireties, by reference thereto.

**[0109]** The Viton® A, Viton® GF, FE5840Q, and FX9038 fluoroelastomers are particularly preferred.

**[0110]** Fluoroelastomer preferably comprises from about 20 percent by volume to about 70 percent by volume of compositions used to prepare coating preparations of the invention. Fluoroelastomer likewise preferably comprises from about 20 percent by volume to about 70 percent by volume of fusing surface layers of the invention.

**[0111]** For preparation of the fusing surface layer, one or more curing agents or curatives are employed in a suitable amount to effect curing of the fluoroelastomer for the continuous phase, and also to effect curing of the discontinuous phase where this phase is in curable form. Suitable curatives for this purpose include nucleophilic addition curing systems. Also appropriate as curatives are free radical initiator curing systems.

**[0112]** Preferred nucleophilic addition curing systems are the bisphenol curing systems. These preferably include at least one bisphenol crosslinking agent and at least one accelerator.

**[0113]** Suitable bisphenol crosslinking agents include 4,4-(hexafluoroisopropylidene)diphenol, also known as bisphenol AF, and 4,4-isopropylidenediphenol. Accelerators which may be employed include organophosphonium salt accelerators such as benzyl triphenylphosphonium chloride.

**[0114]** The amount of bisphenol crosslinking agent used, and likewise the amount of accelerator used, each is preferably from about 0.5 parts to about 10 parts per 100 parts by weight of the fluoroelastomer for the continuous phase. A bisphenol curing system, taken as a whole, is employed in an amount, based on the total weight of crosslinking agent and accelerator, likewise of from about 0.5 parts to about 10 parts per 100 parts by weight of the fluoroelastomer. A commercially available bisphenol curing system which may be used is Viton® Curative No. 50 from Dupont Dow Elastomers, which is a combination of bisphenol AF and one or more quaternary phosphonium salt accelerators; this curative preferably is used in an amount of from about 2 parts to about 8 parts per 100 parts by weight of the fluoroelastomer.

**[0115]** Further nucleophilic addition curing systems are polyfunctional hindered curing systems, particularly diamine curing systems. Among the diamine curing systems that may be employed are diamine carbamate curing systems. Examples of these are hexamethylenediamine carbamate and N,N'-dicinnamylidene-1,6-hexanediamine; these are commercially available as DIAK No. 1 and DIAK No. 3, respectively, from E.I. Du Pont de Nemours, Inc. DIAK No. 4 is another polyfunctional hindered diamine curing system that may be used.



**[0116]** Free radical initiator curing systems which may be used include peroxide free radical initiator curing systems. Preferably these comprise at least one peroxide free radical initiator, and at least one suitable crosslinking agent; peroxides that may be employed for this purpose include the suitable aliphatic peroxides.

**[0117]** Particular peroxides which may be used include ditertiary butyl peroxide, dicumyl peroxide, 2,4-dichlorobenzoyl peroxide, 2,5-dimethyl-2,5-di(t-butyl peroxy)hexane, dibenzoyl peroxide and the like. Particular crosslinking agents suitable for these systems include triallyl cyanurate, triallyl isocyanurate, and others known in the art.

**[0118]** Where the curative comprises a nucleophilic addition curing system or a free radical initiator curing system, one or more cocuratives may also be employed. In this regard, the use of these systems for curing fluoroelastomers can generate hydrogen fluoride. Accordingly, acid acceptors for neutralizing the hydrogen fluoride are suitable cocuratives. Preferred examples of these acid acceptors are the Lewis bases, particularly inorganic bases such as alkali and alkaline earth metal bases. Preferred bases include magnesium oxide, zinc oxide, lead oxide, calcium oxide, and hydroxides including calcium hydroxide, magnesium hydroxide, potassium hydroxide and sodium hydroxide. Hydrides may also be employed including sodium borohydride and lithium aluminum hydride.

**[0119]** Also suitable as a cocurative is the cocurative system disclosed in U.S. Application No. 09/450,302, filed November 29, 1999. This application is incorporated herein in its entirety, by reference thereto.

**[0120]** The amount of cocurative which is used preferably is from about 2 parts to about 20 parts per 100 parts by weight

of the fluoroelastomer. Particularly where one or more acid acceptors are employed, the amount used is preferably that which is sufficient to neutralize the indicated hydrogen fluoride and allow for complete crosslinking.

**[0121]** An excessive amount of cocurative, particularly in the case of the more basic curatives such as calcium hydroxide, will shorten the life of the fluoroelastomer solution or dispersion used to prepare the fusing surface layer, as discussed herein. Specifically, cocurative excess will cause rapid viscosity increase and solution gellation; however, this is not a problem if solution coating is not used.

**[0122]** Magnesium oxide, calcium hydroxide, and zinc oxide are preferred acid acceptors. Particularly for solution coatings, magnesium oxide and zinc oxide are preferred acid acceptors.

**[0123]** A fluoroelastomer composition, such as is used for preparing the fluoroelastomer solution or dispersion of the invention, can comprise the fluoroelastomer for the continuous phase, and also can comprise the material for the discontinuous phase - though, as indicated, this material can be added subsequently to the solvent. The fluoroelastomer composition can additionally include one or more of those of the foregoing curative, cocurative, filler, adjuvant, and additive components that are being employed.

**[0124]** In particular embodiments, this composition can comprise the fluoroelastomer and discontinuous phase material, or the fluoroelastomer and cocurative, or the fluoroelastomer, discontinuous phase material, and cocurative. Any of these embodiments of the fluoroelastomer composition further can include one or more of the curative, filler, adjuvant, and additive components as indicated.

**[0125]** The indicated fluoroelastomer composition may be formed by any means suitable for combining the components. An appropriate dry compounding method is preferred.

**[0126]** Dry compounding may be conducted with a two roll mill. It may be carried out at a temperature of from about 40°F to about 200°F, or from about 50°F to about 100°F. However, preferably the compounding is carried out at approximately room temperature, for example, from about 50°F to about 70°F (from about 10°C to about 21°C), more preferably from about 55°F to about 65°F (from about 13°C to about 28°C). This operation tends to generate heat, so preferably a mill with its operating temperature inhibited by some means, such as by water cooling, is employed. The materials are compounded until a uniform, dry, flexible composite sheet is obtained.

**[0127]** With a fluoroelastomer that, as discussed, is plastic at ambient temperature, the milling operation may be facilitated by raising the temperature at which the compounding is conducted to the softening point of this elastomer. However, with this type of fluoroelastomer, dry compounding may not be practical, in which case combination in solution is preferred. Solvents that are suitable for this purpose include those employed for forming the fusing member layer.

**[0128]** Commercially provided fluoroelastomers often come with curatives already incorporated therein. However, for solution coating it is preferred that the curative not be provided in this manner, but rather be employed as a separate component.

**[0129]** Although curative, as such a separate component, may be dry compounded with the other indicated components, preferably it is not, but rather is subsequently added to the solution or dispersion which is prepared using the dry compounded materials, as discussed herein. Specifically, the

curative may be added directly to the solution or dispersion prior to coating. Withholding the curative thusly for addition to the final coating solution or dispersion greatly extends the shelf life of this solution or dispersion.

**[0130]** For forming the requisite layer on the fuser member, the fluoroelastomer composition can be combined with suitable solvent. Specifically in the case of the fluoroelastomer composition obtained from dry compounding, this composition is divided into pieces and added to a sufficient amount of one or more solvents to provide a solution, or a dispersion.

**[0131]** Further components may also be employed. Although they can be included in the dry compounding, preferably they are added to the solvent. For instance, as indicated, the material for forming the discontinuous phase preferably is added to the solvent - particularly in the case of curable siloxane polymers, and especially where SFR-100 silicone is employed.

**[0132]** Additionally - optionally for dry compounding if employed, but instead preferably added to the solvent - one or more of the polydiorganosiloxane oligomers, particularly the  $\alpha,\omega$  difunctional polydiorganosiloxanes, disclosed in U.S. Patent No. 4,853,737, may be used; this patent is incorporated herein in its entirety, by reference thereto. These polydiorganosiloxanes may be employed in the amount of from about 0.1 to 5 grams per 100 grams of solution.

**[0133]** If both polydiorganosiloxane oligomer and curable siloxane polymer, as discussed, are used, it is preferable that they be kept separate prior to addition to the fluoroelastomer, because these polydiorganosiloxane oligomers catalyze the crosslinking of the curable siloxane polymers.

**[0134]** Still further, one or more yet additional additives and/or adjuvants can be added to the solution, such as

defoaming agents, wetting agents, and other materials. These yet additional adjuvants and fillers, where present, are provided in amounts and proportions as are generally known or as can be determined without undue experimentation by those of ordinary skill in the art.

**[0135]** The amount of solvent used is preferably that which will provide a solution or dispersion having a solids content of from about 10 weight percent to about 50 weight percent, more preferably from about 10 weight percent to about 30 weight percent. Suitable solvents include esters, ketones, and acetates. Ketones that can be used include acetone, methyl ethyl ketone (MEK), and methyl isobutyl ketone. Preferred esters are the C<sub>1</sub>-C<sub>8</sub> acetates, such as the C<sub>2</sub>-C<sub>8</sub> acetates - e.g., ethyl acetate, n-propyl acetate, isopropyl acetate, n-butyl acetate, isobutyl acetate, t-butyl acetate, and sec-butyl acetate. Most preferably the solvent is MEK.

**[0136]** One or more solvents may be employed. Particularly, a mixture of two or more solvents can be used.

**[0137]** For instance, a solvent that can be used is one comprising about 50 weight percent each of methyl ethyl ketone and methyl isobutyl ketone. Yet other solvents which may be used are blends of methyl ethyl ketone and methanol (MeOH), such as blends comprising about 85 percent by weight methyl ethyl ketone and about 15 percent by weight methanol (85:15 MEK:MeOH). Methanol is used to extend the solution life of the coating, or to improve the coating quality.

**[0138]** What is accordingly obtained is a coating composition or preparation - e.g., a coating solution or a coating dispersion - for preparing a fusing surface layer of the invention. With curative being present therein as indicated, it can be designated a curable composition.

**[0139]** The solution or dispersion may be applied to the fuser base in a succession of thin coatings, either as discrete layers or as a continuous buildup of layers. Application is by any suitable means, such as dipping, spraying, or transfer coating.

**[0140]** A method of dipping is ring coating. To conduct ring coating, the roller is drawn up through a larger diameter hole machined in two plates, a top plate and a bottom plate. Between the plates is a flexible gasket which forms a liquid tight seal with the roller surface and the top plate. The coating solution is poured into a well created by the roller, the flexible gasket, and the top plate. The roller is drawn up through the gasket and the solution coats the outside of the roller surface. In this manner a minimal amount of solution is used to coat each roller.

**[0141]** After it is applied, each coating is allowed to stand, at room temperature or higher, in order to flash off all or at least most of the solvent. For instance, following each application of a coating layer, evaporation of solvent is effected at temperatures of from about 25°C to about 90°C or higher.

**[0142]** When the desired thickness is obtained the resulting layer is cured. Preferably, the layer is heated to a temperature of from about 150°C to about 250°C and held for 12 to 48 hours. To prevent bubbling of the layer, either sufficient drying time is allowed for the indicated solvent flash off or evaporation to be completed, or the ramp to cure temperature - i.e., from room temperature to the stated 150°C-250°C upper limit - is extended over a period of 2 to 24 hours.

**[0143]** The number of coatings applied to form the fusing surface layer is that which will provide the appropriate

thickness, which can be within a range as is conventional in the art. Specifically, the fusing surface layer can be of a thickness as is suitable for the systems and processes in which it is employed, and the requisite thickness for particular instances can be determined without undue experimentation.

**[0144]** Further as to fusing surface layer thickness, one factor to consider, with respect to the acceptable minimum thickness, is whether there is a cushion interposed between the fusing surface layer and fuser base. The presence of an intermediate compliant layer allows for stretching of the fusing surface layer during use. Accordingly, in addition to normal wear that is occurring, the delamination effect acting on the fusing surface layer is magnified. And the thicker the cushion interposed between fusing surface layer and base, the more this effect is magnified.

**[0145]** As to the foregoing, where the fusing surface layer of the invention resides right on the fuser base, then there is no deformability to magnify the delamination effect. In this instance, the fusing surface layer can be as thin as about 12 microns.

**[0146]** However, where there is a cushion layer or layers interposed between fusing surface layer and base, then if the total thickness of the intermediate compliant layer or layers is less than or equal to about 2500 microns, the surface layer should have a thickness of not less than about 25 microns. And if the total thickness of the intermediate compliant layer or layers is greater than about 2500 microns, then the fusing surface layer should be at least about 38 microns thick.

**[0147]** There are also factors to consider as to preferred maximum fusing surface layer thicknesses in various circumstances. For instance, if internal heating is employed

in the fusing process , then the fusing surface layer must not be so thick as to impede heat transfer impermissibly, and thereby cause the base or core temperature to become excessive. Accordingly, even where the fusing surface layer is directly adjacent to the base, the layer preferably is not thicker than about 400 microns.

**[0148]** Where there is no internal heating, then the fusing surface layer can be thicker. In these circumstances the fusing surface layer can be as thick as about 1000 microns, or even thicker; theoretically there is no thickness upper limit, subject to considerations of cost and processing limitations.

**[0149]** However, if the fusing surface layer of the invention is being provided by a solution or dispersion coating method, such as the method discussed herein, then this factor is likely to put a practical upper limit on thickness. Because of the restricted amount of coating deposited by each application, then a point is reached where multiple iterations become operationally difficult. Accordingly, solution or dispersion coating methods as are known generally limit the surface layer to a thickness of about 500 microns or less.

**[0150]** The fusing surface layer of the invention may be provided by methods, other than solution coating, which are suitable. For instance, appropriate extrusion coating methods may be used.

**[0151]** Toners suitable for the process of the invention include linear toners and branched toners. With branched toners, the branching may be inherent in the toner polymer, or generated using crosslinking agents. Correspondingly, toners that may be used include those characterized by partial crosslinking, with the degree of crosslinking being that which increases toner viscosity, but which is not sufficient to provide a fully crosslinked rubber.



**[0152]** Preferably the toners used are those having a  $\tan \delta$  of less than about 1.6. As a matter of particular preference, the toners used are those having a  $\tan \delta$  of from about 0.4, or about 0.5, or about 0.6, to about 1.0, or about 1.2, or about 1.5.

**[0153]** The viscosity of a toner employed in the process of the invention must be sufficiently high at fuser operating conditions, or as discussed herein, at the temperature of the fusing process. If, in the fusing process, the toner viscosity is too low, the compression of the toner in the fusing process seemingly will be too rapid, and therefore fail to generate the variable height - and particularly, fail to generate sufficient height variation -in the toner surface for providing the desired low gloss.

**[0154]** Accordingly, at the requisite conditions as indicated, the toner has a viscosity preferably of at least about 10 Kpoise, more preferably of at least about 40 Kpoise, and still more preferably of at least about 100 Kpoise.

**[0155]** Conventional toners meeting the viscosity requirements as stated may be employed. Suitable toners include styrene-butadiene, styrene-butylacrylate, and polyester thermoplastic toners. A particular toner which may be used is a partially crosslinked styrene-butylacrylate toner having, at the temperature of the fusing process, a viscosity of at least about 100 Kpoise. Another particular toner which may be used is a partially crosslinked polyester toner having, at the temperature of the fusing process, a viscosity of at least about 40 Kpoise.

**[0156]** In the operation of the toner fusing system of the invention, release agent can be applied to the fusing surface layer so that this agent contacts toner on the substrate, and can also contact the substrate, during the operation of the

fuser member. Particularly where the fuser base is a cylindrical roller or an endless belt, the release agent is applied, while the base is rotating or the belt is running, upstream of the contact area between fuser member and substrate toner.

**[0157]** If employed, release agent preferably is applied so as to form a film on the fusing surface layer. As a matter of particular preference, the release agent is applied so as to form a film that completely, or at least essentially or at least substantially, covers the fusing surface layer. Also as a matter of preference, during operation of the system the release agent is applied continuously, or at least essentially or at least substantially continuously, to the fusing surface layer.

**[0158]** Release agents are intended to prohibit, or at least lessen, offset of toner from the substrate to the fusing surface layer. In performing this function, the release agent can form, or participate in the formation of, a barrier or film that releases the toner. Thereby the toner is inhibited in its contacting of, or even prevented from contacting, the actual fusing surface layer, or at least the fluoroelastomer thereof.

**[0159]** The release agent can be a fluid, such as an oil or a liquid, and is preferably an oil. It can be a solid or a liquid at ambient temperature, and a fluid at operating temperatures. Also as a matter of preference, the release agent is a polymeric release agent, and as a matter of particular preference, is a silicone or polyorganosiloxane oil.

**[0160]** Suitable release agents are those disclosed in U.S. Patents Nos. 5,824,416, and 5,780,545. These two patents are incorporated herein in their entireties, by reference thereto.

**[0161]** Further as to this matter, release agents which may be used include polymeric release agents having functional groups. Appropriate polymeric release agents with functional groups include those which may be found as liquids or solids at room temperature, but are fluid at operating temperatures.

**[0162]** Particular functional group polymeric release agents which may be used include those disclosed in U.S. Patents Nos. 4,011,362, 4,046,795, and 5,781,840; these patents also are incorporated herein in their entireties, by reference thereto. Still further release agents which may be used are the mercapto functional polyorganosiloxanes disclosed in U.S. Patent No. 4,029,827, and the polymeric release agents having functional groups such as carboxy, hydroxy, epoxy, amino, isocyanate, thioether, and mercapto functional groups, as disclosed in U.S. Patents Nos. 4,101,686 and 4,185,140; yet additionally these patents are incorporated herein in their entireties, by reference thereto.

**[0163]** Preferred release agents with functional groups include the mercapto functional polyorganosiloxane release agents and the amino functional polyorganosiloxane release agents. Particularly preferred are the release agents, including mercapto functional polyorganosiloxane release agents, consisting of, consisting essentially of, consisting substantially of, or comprising monomercapto functional polyorganosiloxanes, or polyorganosiloxanes having one mercapto functional group per molecule or polymer chain. Also particularly preferred are release agents, including amino functional polyorganosiloxane release agents, consisting of, consisting essentially of, consisting substantially of, or comprising monoamino functional polyorganosiloxanes, or polyorganosiloxanes having one amino functional group per molecule or polymer chain. In this regard, the release agents

disclosed in U.S. Patents Nos. 5,531,813 and 6,011,946 may be used; these patents are incorporated herein in their entireties, by reference thereto.

**[0164]** Additional preferred release agents are the fluoro functional polyorganosiloxanes, including those with fluoroalkyl, such as trifluoroalkyl (e.g. trifluoropropyl) functionality, and fluorosilicones, and polyorganosiloxanes having fluorine-containing groups, as disclosed in U.S. Patents Nos. 5,568,239, 5,627,000, and 5,641,603; these patents also are incorporated herein in their entireties, by reference thereto. The fluoro functional polyorganosiloxanes are particularly preferred where the fusing surface layer comprises fluoroplastic and/or fluoro resin particles.

**[0165]** Further with regard to the functional agents, one point to consider is that because of their expense usually they are diluted with nonfunctional polyorganosiloxanes, particularly nonfunctional polydimethylsiloxanes. Another point is that for obtaining good release activity with a functional release agent, monofunctionality is preferred, so that the molecule cannot react both with toner and with the fusing surface layer, and thereby serve as a toner/fuser member adhesive. Therefore, the functional agent would ideally consist of entirely, or at least consist essentially, of the monofunctional moiety. However that also is impractical, also because of expense.

**[0166]** Therefore, the functional polyorganosiloxane preferably comprises as great a proportion of the monofunctional moiety as is practically possible. As a matter of particular preference, the functional polyorganosiloxane has a sufficient monofunctional proportion so as not to act as the indicated adhesive.

**[0167]** Accordingly, a preferred release agent composition comprises a blend of nonfunctional polyorganosiloxane, particularly nonfunctional polydimethylsiloxane, with amino functional polyorganosiloxane, and the amino functional polyorganosiloxane comprises monoamino functional polyorganosiloxane. Another preferred release agent composition comprises a blend of nonfunctional polyorganosiloxane, particularly nonfunctional polydimethylsiloxane, with mercapto functional polyorganosiloxane, and the mercapto functional polyorganosiloxane comprises monomercapto functional polyorganosiloxane.

**[0168]** The release agent may be applied to the fuser member by any suitable applicator, including sump and delivery roller, jet sprayer, etc. Those means as disclosed in U.S. Patents Nos. 5,017,432 and 4,257,699 may be employed; the latter of these two patents is incorporated herein in its entirety, by reference thereto. Preferably the present invention employs a rotating wick oiler or a donor roller oiler.

**[0169]** A rotating wick oiler comprises a storage compartment for the release agent and a wick for extending into this compartment. During operation of the toner fusing system of the invention, the wick is situated so as to be in contact with the stored release agent and also with the fusing surface layer of the fuser member; the wick thusly picks up release agent and transfers it to the fuser member.

**[0170]** A donor roller oiler includes two rollers and a metering blade, which can be a rubber, plastic, or metal blade. One roller meters the oil in conjunction with the blade, and the other transfers the oil to the fuser roller. This type of oiler is common in the art, and is frequently

used with fuser members having fluoroelastomer fusing surface layers.

**[0171]** The release agent is applied to the substrate, particularly in the case of paper, preferably at a rate of from about 0.1 to about 20 microliters, more preferably at a rate of about 1.0 to about 8 microliters, per 8½" by 11" copy. The applicator accordingly is adjusted to apply the release agent at this rate.

**[0172]** Images characterized by the requisite low gloss are obtained by operation of the toner fusing process of the invention so that multiple particular process conditions are met.

**[0173]** In this regard, there has to be a sufficient amount of the discontinuous phase material in the fusing surface layer. Specifically, the discontinuous phase must comprise at least a minimum proportion by volume of the fusing surface layer, and/or at least a minimum proportion by weight of the fluoroelastomer continuous phase.

**[0174]** Further, at the fusing temperature, not only must the modulus of the fluoroelastomer continuous phase be greater than the modulus of the discontinuous phase domains, but also there must be a sufficient difference between the modulus of the continuous phase and the modulus of the discontinuous phase. The difference may be relatively small, such as the difference between a fluoroelastomer continuous phase having a modulus of  $8 \times 10^6$  Pa, and a discontinuous phase having a modulus of  $0.8 \times 10^6$  Pa; or relatively large, as in the case of a continuous phase having a modulus of  $5 \times 10^8$  Pa, and a discontinuous phase having a modulus of  $0.5 \times 10^6$  Pa.

**[0175]** In the first of the above two examples, the continuous phase modulus of  $8 \times 10^6$  Pa may be expressed as the logarithm value of about 6.9, and the discontinuous phase modulus of 0.8

$\times 10^6$  Pa may be expressed as the logarithm value of about 5.9; the difference between these two logarithm values is about 1.0. In the second example, the continuous phase modulus of  $5 \times 10^8$  Pa may be expressed as the logarithm value of about 8.69897, and the discontinuous phase modulus of  $0.5 \times 10^6$  Pa may be expressed as the logarithm value of about 5.69897; the difference between these two logarithm values is about 3.0.

**[0176]** Thus, and more generally, the difference between the logarithms of the moduli of the two respective phases is preferably at least about 1.0, more preferably at least about 1.4, and still more preferably between about 1.4 and about 3.0.

**[0177]** Yet additionally, at the fusing temperature the viscosity of the toner must, as indicated, be high enough. Particularly, the viscosity of the toner preferably is high enough so that the toner sufficiently indents the discontinuous phase domains that contact the toner - i.e., exposed domains, at the top of the fusing surface layer.

**[0178]** Correspondingly, at the fusing temperature the modulus of the discontinuous phase domains must be low enough. Particularly, the modulus of the discontinuous phase domains preferably is low enough so that the domains as indicated are sufficiently indented by the toner.

**[0179]** Also, the amount of heat transmitted to the toner, in the fusing process, must be sufficient to fuse the toner to the substrate, but not enough to bring image gloss above the intended maximum level.

**[0180]** Of the foregoing conditions, those pertaining to modulus - i.e., the continuous phase modulus being greater than the discontinuous phase modulus, and the difference between the moduli being sufficient, and the continuous phase

modulus being low enough - particularly are met where the fusing surface layer is at its equilibrium surface.

**[0181]** As to quantifiable values and/or value ranges for process conditions that, in actual practice of the fusing process, will provide gloss at an intended level - e.g., a gloss number of about 10 or less, or about 8 or less, or about 6 or less, or about 5 or less - combinations of the requisite values and/or ranges can be determined. And in fact, the numerical values and/or ranges for particular conditions will depend generally on the operating conditions of the process, including the numerical values and/or value ranges of the other conditions.

**[0182]** Where the process of the invention generates an image having a gloss number of about 10 or less, preferably the domains of the discontinuous phase have a mean diameter of at least about 0.5 microns. Where the process of the invention generates an image having a gloss number of about 8 or less, preferably the domains of the discontinuous phase have a mean diameter of at least about 1 micron. Where the process of the invention generates an image having a gloss number of about 6 or less, preferably the domains of the discontinuous phase have a mean diameter of at least about 2 microns. Where the process of the invention generates an image having a gloss number of about 5 or less, preferably the domains of the discontinuous phase have a mean diameter of at least about 4 microns.

**[0183]** Where the process of the invention generates an image having a gloss number of about 10 or less, preferably the fusing surface layer comprises at least about 20 parts discontinuous phase per 100 parts by weight of the fluoroelastomer continuous phase. Where the process of the invention generates an image having a gloss number of about 8



or less, preferably the fusing surface layer comprises at least about 30 parts discontinuous phase per 100 parts by weight of the fluoroelastomer continuous phase. Where the process of the invention generates an image having a gloss number of about 6 or less, preferably the fusing surface layer comprises at least about 40 parts discontinuous phase per 100 parts by weight of the fluoroelastomer continuous phase. Where the process of the invention generates an image having a gloss number of about 5 or less, preferably the fusing surface layer comprises at least about 50 parts discontinuous phase per 100 parts by weight of the fluoroelastomer continuous phase.

**[0184]** Where the process of the invention generates an image having a gloss number of about 10 or less, the discontinuous phase preferably comprises at least about 10 percent by volume, more preferably at least about 15 percent by volume, and still more preferably at least about 20 percent by volume, of the fusing surface layer. Where the process of the invention generates an image having a gloss number of about 8 or less, the discontinuous phase preferably comprises at least about 20 percent by volume, more preferably at least about 25 percent by volume, and still more preferably at least about 30 percent by volume, of the fusing surface layer. Where the process of the invention generates an image having a gloss number of about 6 or less, the discontinuous phase preferably comprises at least about 25 percent by volume, more preferably at least about 30 percent by volume, and still more preferably at least about 35 percent by volume, of the fusing surface layer. Where the process of the invention generates an image having a gloss number of about 5 or less, the discontinuous phase preferably comprises at least about 30 percent by volume, more preferably at least about 35 percent by volume,

and still more preferably at least about 40 percent by volume, of the fusing surface layer.

**[0185]** As the fuser member is used during the fusing process, the fusing surface layer will provide an equilibrium surface. This equilibrium surface will be provided either from the beginning of use, or some point during the course of use, as the layer is being worn away.

**[0186]** Taking the fusing surface layer at a cross section - i.e., starting at the top, or surface, of the layer, and extending downwardly toward the fuser base to the bottom of this layer - the equilibrium surface is provided by that portion of the layer which is homogeneous, or at least essentially homogeneous, in composition. Particularly, in the portion of the fusing surface layer, taken at the indicated cross section, which provides the equilibrium surface, all the components of the layer are distributed uniformly, or at least essentially uniformly.

**[0187]** At the equilibrium surface of the fusing surface layer, the components of the surface, the proportions of these components, the distribution of these components, and the roughness of the surface remains unchanged, or at least essentially unchanged, as the use of the fuser member in the fusing process proceeds. At its equilibrium surface, the wearing away of the fusing surface layer, at its surface, regenerates a surface wherein the indicated features remain the same, or at least essentially the same.

**[0188]** And with roughness as indicated remaining the same, or at least essentially the same, at the equilibrium surface the fusing surface layer is accordingly also at equilibrium surface roughness. Particularly, equilibrium surface roughness is an aspect of equilibrium surface.

**[0189]** Preferably in the process of the invention, at the equilibrium surface of the fusing surface layer:

- the modulus of the fluoroelastomer continuous phase is greater than the modulus of the discontinuous phase at the temperature of the fusing process;

- the difference between the modulus of the fluoroelastomer continuous phase and the modulus of the discontinuous phase is sufficiently great at the temperature of the fusing process;

- the modulus of the discontinuous phase is sufficiently low at the temperature of the fusing process; and

- the discontinuous phase comprises at least a minimum proportion by volume of the fusing surface layer, and/or at least a minimum proportion by weight of the fluoroelastomer continuous phase;

so that the image generated in the process has a gloss number of about 10 or less - more preferably of about 8 or less, still more preferably of about 6 or less, and still more preferably of about 5 or less.

**[0190]** Where the fusing surface layer of the invention is not already at its equilibrium surface at the top of the layer, or at the beginning or outset of use in the fusing process, then during use the layer wears away to the point at which it reaches its equilibrium surface. The particular method disclosed herein, for preparing the fusing surface layer, results in a layer that is characterized by homogeneity, or at least essential homogeneity, throughout the layer, except at the very top. And the fusing surface layer correspondingly provides an equilibrium surface throughout the layer, except at the very top.

**[0191]** Specifically, this method of preparation leaves the layer with an uppermost portion, or artifact, which is not at

equilibrium surface. When this artifact is worn away, such as by use in the fusing process, the fusing surface layer is at its equilibrium surface.

**[0192]** In fact - rather than being left with the artifact to be worn away by use - the fusing surface layer obtained from the preparation method disclosed herein can be subjected to a surface finishing treatment, so that it indeed does exhibit equilibrium surface at its very top, or at the beginning of use. For instance, the artifact may be removed from the layer prior to use.

**[0193]** Where the fusing surface layer is not thusly at its equilibrium surface from the outset, it nevertheless may - depending upon its composition - generate an image at or below the requisite maximum gloss prior to the equilibrium being reached; it even may be that the sufficiently low degree of gloss is generated at the beginning of use and continuing thereafter - i.e., is provided by the very top of the layer, and still provided as the layer wears away - even where the layer is not yet at its equilibrium surface. Nevertheless, regardless of whether the layer is producing the gloss as indicated before the equilibrium surface is reached, it does so at this equilibrium.

**[0194]** Specifically as to the amount of heat employed in the fusing process, it is noted, with respect to this process condition, that the heat flowing into the toner causes its viscosity to drop. The amount of fusing process heat therefore can affect the gloss of the image ultimately generated.

**[0195]** The amount of heat that is employed is a function of both the time that toner and its supporting substrate reside in the nip, and also the temperature of the fusing process,

and particularly of the fusing surface layer contacting the toner.

**[0196]** Accordingly, increasing or decreasing either or both of these factors respectively increases or decreases the amount of heat employed in the fusing process. And correspondingly, one or both of nip residence time and fusing temperature can be adjusted to affect the resultant gloss level - and even to achieve a desired maximum gloss level, such as a gloss number of about 10 or less, or about 8 or less, or about 6 or less, or about 5 or less - of the image obtained from the fusing process.

**[0197]** Preferably the nip residence time is from about 12 to about 22 milliseconds. Also as a matter of preference, the temperature of the fusing process, and preferably particularly of the fusing surface layer contacting the toner, is from about 120°C to about 200°C.

**[0198]** It is thought that the desired low gloss of the generated image results from the configuration, of the surface of the fused toner, provided by the fusing process. And it is also thought that this configuration is caused by the toner indenting discontinuous phase domains of the fusing surface layer.

**[0199]** In this regard, it is believed that, during the application of pressure to the toner by the fusing surface layer, the toner gives greater resistance to the discontinuous phase domains than to the continuous phase. Where both phases are compressing the toner, it is believed that they do so at different rates - with compression by the continuous phase seemingly occurring at a faster rate than compression by the domains, because of the differing hardness between discontinuous phase domains and the continuous phase, and because the viscosity of the toner is high enough so that its

compression by the two different phases is sufficiently uneven. Particularly, the viscosity of the toner is high enough so that the difference, in the rate of compression effected by the two different phases, is sufficiently great for the purpose of fusing process.

**[0200]** The result is thought to be that, as ascertained at the interface of toner and fusing surface layer, the toner indents domains with which it is in contact, with a variable height pattern being provided to the toner surface. And this unevenness is thought to produce desired low gloss.

**[0201]** The foregoing is provided as the best explanation available, for the mechanisms involved in the fusing process, as they are currently understood. It is presented so as to discuss the invention as fully as possible according to this best current understanding, and is not to be considered as limiting the scope of the invention.

**[0202]** A toner fusing system of the invention is shown in Fig. 1. Multilayered fuser roller 10 comprises, in sequential order, a fuser base 11, in the form of a hollow cylindrical roller, as well as a cushion layer 12 and a fusing surface layer 13. Fusing surface layer 13 has silicone elastomer particulate(not depicted in Fig. 1) dispersed therein. Internal heating member 14, an optional element in the invention, is disposed in the hollow portion of fuser base 11.

**[0203]** External heating members 15 and 16 are in the form of hollow cylindrical rollers; their rotational directions, and the rotational directions of all the other rotating elements, is shown by their respective arrows. The rotational directions as depicted can all be reversed.

**[0204]** External heating members 15 and 16 are heated by respective heating lamps 17. These two contact heating members are spaced apart by a distance less than the diameter

of fuser member 10, which is in contact with both. Contact heating members 15 and 16 transfer heat to fuser member 10 by their contact with fusing surface layer 13.

**[0205]** Rotating wick oiler 18 applies release agent to fusing surface layer 13.

**[0206]** Support member 19, in the form of a backup roller, cooperates with fuser member 10 to form fusing nip or contact arc 20. Copy paper or other substrate 21, carrying unfused toner images 22, passes through fusing nip 20 so that toner images 22 are contacted by fusing surface layer 13. Support member 19 and fuser member 10 act together to apply pressure to the paper 21 and toner 22, and fuser member 10 also provides heat, with the heat and pressure serving to fuse toner 22 to the paper 21.

**[0207]** Dispensing roller 26 incrementally feeds cleaning web 24 over advance roller 25, to be rolled up onto collecting roller 23. In passing along roller 25, web 24 contacts and cleans contact heating members 15 and 16.

**[0208]** Cleaning web 24 is a polyamide material. A polyamide web which may be employed for this purpose is commercially available under the trademark Nomex ® from BMP of America, Medina, NY. Any other suitable cleaning material may be employed instead.

**[0209]** In place of the indicated cleaning assembly, any other means or apparatus appropriate for cleaning the contact heating members may be employed. Alternatively, the contact heating members can be provided with a nonstick coating. This coating can be a fluoroplastic, and it can include a heat conducting filler. Where the contact heating members have a nonstick coating the means for cleaning these members can be omitted.

**[0210]** Fig. 2 shows a fragmentary view of an embodiment of fuser member 10, magnified to show the multiple layers in greater detail. Silicone elastomer particles 27 are distributed through fusing surface layer 13.

**[0211]** Fig. 3 shows a fragmentary view of another embodiment of fuser member 10, also magnified to show greater detail. In this embodiment there is no cushion, and fusing surface layer 13 resides directly on fuser base 11.

**[0212]** The invention is illustrated by the following procedures; these are provided for the purpose of representation, and are not to be construed as limiting the scope of the invention. Unless stated otherwise, all percentages, parts, etc. are by weight.

#### EXPERIMENTAL PROCEDURES

##### Materials Employed in the Procedures

**[0213]** Viton® A fluoroelastomer, a copolymer of vinylidene fluoride and hexafluoropropylene

**[0214]** Fe<sub>2</sub>O<sub>3</sub> (7098), approx. 0.7 microns mean particle diameter, from Harcros Pigments Inc., Easton, PA

**[0215]** Al<sub>2</sub>O<sub>3</sub> (AL7131), approx. 5 microns mean particle diameter, from Norton Materials, Worcester, MA

**[0216]** Al<sub>2</sub>O<sub>3</sub> (T-64), approx. 12 microns mean particle diameter, from Whitaker Clark & Daniels, Inc., South Plainfield, NJ

**[0217]** Carbon black (Thermax), from R.T. Vanderbilt Company Inc., Norwalk, CT

**[0218]** Dow™ 1200 RTV Prime Coat primer, from Dow Corning Corporation. A metal alkoxide type primer containing light aliphatic petroleum naphtha (85 weight percent), tetra(2-methoxy-ethoxy)silane (5 weight percent), tetrapropyl orthosilicate (5 weight percent), and tetrabutyl titanate (5 weight percent)



**[0219]** Silastic™-J 60 Shore A addition cure RTV silicone rubber, from Dow Corning Corporation

**[0220]** EC4952 65 Shore A condensation cure RTV silicone rubber, from Emerson & Cuming ICI

**[0221]** PS513  $\alpha,\omega$ 3-aminopropyldimethylsiloxyl terminated polydimethylsiloxane, from United Chemical Technologies, Inc., Bristol, PA

**[0222]** 60,000 centistoke DC200 polydimethylsiloxane, from Dow Corning Corporation

**[0223]** MgO (Maglite™-Y), from Merck/Calgon Corp., Teterboro, NJ

**[0224]** Viton® Curative No. 50, from Dupont Dow Elastomers

**[0225]** Catalyst 50, from Emerson & Cuming ICI

**[0226]** SFR-100 silicone, from GE Silicones

#### Preparation of Fluoroelastomer Compositions

**[0227]** Viton® A and MgO, in amounts as set forth in Table 1, and filler, of the types and in the amounts as also identified in Table 1, were thoroughly compounded on a water cooled two roll mill at 63°F (17°C). For each composition, compounding was conducted until a uniform, dry composite sheet was obtained. The sheet was removed and stored until used for the preparation of a coating solution.

**TABLE 1**

Composition No.	Viton®A (grams)	MgO (grams)	Filler	
			(type)	(grams)
1	400	48	Fe <sub>2</sub> O <sub>3</sub>	664
2	300	36	Al <sub>2</sub> O <sub>3</sub> (AL7131)	123.6
3	500	60	Carbon Black	5
4	200	24	Al <sub>2</sub> O <sub>3</sub> (T-64)	82.4
5	300	36	Al <sub>2</sub> O <sub>3</sub> (T-64)	174.9

Preparation of Fuser Members

**[0228]** The foregoing fluoroelastomer compositions were used to prepare the fuser rollers of Comparative Examples 1-4 and Examples 1-7 in the manner as set forth below.

Comparative Example 1

**[0229]** A cylindrical stainless steel fuser core was cleaned with dichloromethane and dried. The core was then primed with a uniform coat of Dow™ 1200 RTV Prime Coat primer. Silastic™-J silicone rubber part A and B was then mixed, injection molded onto the core, and cured at 232°C for 2 hours under 75 tons/inch<sup>2</sup> of pressure.

**[0230]** The roller was then removed from the mold and baked in a convection oven with a temperature ramp increasing to 232°C substantially uniformly over 24 hours, and this temperature then being maintained for an additional 24 hours. After air cooling, EC4952 silicone rubber was blade coated directly onto the Silastic™-J silicone rubber layer, then cured for 12 hours at about 210°C, followed by 48 hours at 218°C in a convection oven. After air cooling, the EC4952 silicone layer was ground to a thickness of 0.457 mm (0.018 inches), and the thusly layered fuser core was corona discharge treated for 1 minute at 300 watts.

**[0231]** The resulting product was a fuser core with a cushion made up of a Silastic™-J silicone layer having a thickness of 4.572 mm (0.180 inches), overlaid by an EC4952 silicone layer having the thickness as indicated. To prepare for coating the fluoroelastomer fusing surface layer thereon, the cushion was wiped with isopropyl alcohol.

**[0232]** A fluoroelastomer solution was prepared by dividing 80 grams of Composition 1 into pieces, and placing this material in a glass jar with 120 grams of MEK, to dissolve the

Composition 1 material in the MEK. The jar was sealed, placed on its side on the indicated roll mill, and rotated to effect gentle stirring. For subsequent addition of materials - in this Comparative Example, and in the following Examples and Comparative Examples - the jar was taken off the roll mill, unsealed, addition was effected, the jar was again placed on its side on the mill, and rotation to provide gentle stirring was resumed. This stirring was continued until initiation of ring-coating, as discussed below.

**[0233]** Specifically for this Comparative Example, 0.83 grams of PS513 and 1.63 grams of Viton® Curative No. 50 were added to the solution. 30 minutes after addition of the PS513 and the Viton® Curative No. 50, the fluoroelastomer solution was ring-coated onto the corona discharge treated roller thrice, allowing the coating to dry between coats. The thusly-coated roller was cured by ramping the temperature from room temperature to 230°C over a 12 hour period, and then holding the temperature at 230°C for 24 hours.

**[0234]** The thickness of the fluoroelastomer coating was measured by removing a small portion of the roller surface and measuring the layer thickness by optical microscopy. By this method, the coating was determined to be 3.9 mils thick.

#### Comparative Example 2

**[0235]** A fuser roller was prepared in substantially the same manner as that of Comparative Example 1, except with 60 grams of Composition 2 in place of Composition 1, and with 140 grams of MEK, 0.9 grams of PS513, and 2.72 grams of Viton® Curative No. 50, instead of the amounts specified in Example 1. The thickness of the fluoroelastomer coating was measured by the same manner as in Comparative Example 1, and determined to be 3.78 mils thick.

Comparative Example 3

**[0236]** A fuser roller was prepared in substantially the same manner as that of Comparative Example 1, except with 17.3 grams of Composition 3 in place of Composition 1, and with 72.85 grams of MEK, 0.4 grams of PS513, and 1.47 grams of Viton® Curative No. 50, instead of the amounts specified in Example 1. The thickness of the fluoroelastomer coating was measured by the same manner as in Comparative Example 1, and determined to be 2.56 mils thick.

Comparative Example 4

**[0237]** A fuser roller was prepared in substantially the same manner as that of Comparative Example 1, except with 14.3 grams of Composition 3 in place of Composition 1, and with 60.7 grams of MEK, 0.4 grams of PS513, and 0.39 grams of Viton® Curative No. 50, instead of the amounts specified in Example 1. The thickness of the fluoroelastomer coating was measured by the same manner as in Comparative Example 1, and determined to be 4.37 mils thick.

Example 1

**[0238]** A fuser roller was prepared in substantially the same manner as that of Comparative Example 1, except for the following differences. Specifically, 40 grams of Composition 3 were employed instead of 80 grams of Composition 1. Further, 150 grams of MEK, 1.8 grams of PS513, and 0.4 grams of Viton® Curative No. 50 were employed, instead of the amounts of these materials as specified in Example 1. Yet additionally, in place of adding the PS513 and Viton® Curative No. 50 30 minutes before ring coating, and instead of adding the Viton® Curative No. 50 to the full amount of the solution previously prepared, the following sequence was

employed, with the amounts as indicated: the 1.8 grams of PS513 were added to the solution of Composition 3 in MEK; 24 hours after this addition of PS513, 17.7 grams of SFR-100 were added; 7 hours after addition of the SFR-100, a 97 grams portion of the solution was taken, and the Viton® Curative No. 50 was added to this portion; and the ring coating was conducted 30 minutes after addition of the Viton® Curative No. 50. The thickness of the fluoroelastomer coating was measured by the same manner as in Comparative Example 1, and determined to be 2.8 mils thick.

#### Example 2

**[0239]** A fuser roller was prepared in substantially the same manner as that of Example 1, except the amount of Viton® Curative No. 50 employed was 1.26 grams, and that the amount of solution taken after addition of the SFR-100 was 98 grams, with the indicated Viton® Curative No. 50 being added to this portion. The thickness of the fluoroelastomer coating was measured by the same manner as in Comparative Example 1, and determined to be 3.74 mils thick.

#### Example 3

**[0240]** A fuser roller was prepared in substantially the same manner as that of Example 1, except for the following differences. Specifically, 65 grams of Composition 4 were employed in place of 40 grams of Composition 3, and 155 grams of MEK, 2.123 grams of PS513, 2.95 grams of Viton® Curative No. 50, and 21.23 grams of SFR100 were employed in place of the amounts described in Example 1. Additionally, after addition of the SFR-100, the Viton® Curative No. 50 was added to the entire amount of solution. The thickness of the

fluoroelastomer coating was measured by the same manner as in Comparative Example 1, and determined to be 4.64 mils thick.

#### Example 4

**[0241]** A fuser roller was prepared in substantially the same manner as that of Example 1, except for the following differences. Specifically, 36 grams of Composition 5 were employed in place of 40 grams of Composition 3, and 84 grams of MEK, 0.83 grams of PS513, 1.526 grams of Viton® Curative No. 50, and 10.8 grams of SFR100 were employed in place of the amounts described in Example 1. Additionally, after addition of the SFR-100, the Viton® Curative No. 50 was added to the entire amount of solution. The thickness of the fluoroelastomer coating was measured by the same manner as in Comparative Example 1, and determined to be 4.3 mils thick.

#### Example 5

**[0242]** A fuser roller was prepared in substantially the same manner as that of Example 1, except for the following differences. Specifically, 36 grams of Composition 4 were employed in place of 40 grams of Composition 3, and 84 grams of MEK, 0.216 grams of PS513, 0.526 grams of Viton® Curative No. 50, and 2.16 grams of SFR100 were employed in place of the amounts described in Example 1. Additionally, after addition of the SFR-100, the Viton® Curative No. 50 was added to the entire amount of solution. The thickness of the fluoroelastomer coating was measured by the same manner as in Comparative Example 1, and determined to be 3.35 mils thick.

#### Example 6

**[0243]** A fuser roller was prepared in substantially the same manner as that of Example 5, except with 0.432 grams of PS513

and 4.32 grams of SFR100 employed in place of the amounts described in Example 5. The thickness of the fluoroelastomer coating was measured by the same manner as in Comparative Example 1, and determined to be 4.25 mils thick.

#### Example 7

**[0244]** A fuser roller was prepared in substantially the same manner as that of Example 6, except with 0.648 grams of PS513 and 6.48 grams of SFR100 employed in place of the amounts described in Comparative Example 5. The thickness of the fluoroelastomer coating was measured by the same manner as in Comparative Example 1, and determined to be 3.46 mils thick.

#### Fuser Roller Testing

**[0245]** To compare the respective performances of the fuser rollers of the Comparative Examples and Examples, these rollers were each employed with a Heidelberg Digimaster™ 9110 (HD9110) electrophotographic fusing system. In every instance unfused toner was applied to a paper substrate in the HD9110 system, with the roller being employed in the fixing of the toner to the paper.

**[0246]** The release oil of the HD9110 fuser was changed from the standard 60,000 cSt release fluid to a blend of 87.5 weight percent DC200 and 12.5 weight percent of an  $\alpha$ -3-aminopropyldimethylsiloxyl,  $\omega$ -trimethylsiloxyl terminated polydimethylsiloxane with a number average molecular weight of 12,000. The rate of application was 2.0 milligrams per copy. Otherwise, all materials, hardware and set points used to compare the indicated fuser rollers were consistent with the Heidelberg Digimaster™ 9110 system.

**[0247]** Each roller was placed in the fuser, and the HD9110 system was run with standard 20# bond paper using a variety of

toned images. At print count measurements of about 10,000, about 200,000, and about 300,000, special short runs were employed for conducting gloss level tests; in this regard, the tests at about 10,000 prints actually were conducted between 0 prints and 10,000 prints, while the tests at about 200,000 prints and at about 300,000 prints were conducted within about 20,000 prints of the count - i.e., between about 180,000 and about 220,000 prints, and between about 280,000 and about 320,000 prints, respectively.

**[0248]** The fuser rollers of Comparative Examples 1-4 and Examples 1-7 were tested for gloss level of the generated image. Gloss was measured in terms of gloss number, using the procedure and devices as discussed herein for determining this value. The values obtained are set forth in Table 2 below.

**TABLE 2**

	Particle size*	Filler level**	SFR-100***	Cure Level****	Gloss Number		
					0-10K	200K	300K
Comp.Ex.1	0.7	35	0	2.5	19	18	16.5
Comp.Ex.2	5.0	15	0	2.5	14	14.5	15
Comp.Ex.3	NA	0	0	2.5	7.3	7	7.6
Comp.Ex.4	NA	0	0	0.8	9	9.5	10.3
Ex.1	NA	0	50	2.5	7.1	4.8	-
Ex.2	NA	0	50	0.8	3.6	5	-
Ex.3	12	15	50	2.5	5.7	4.7	4.9
Ex.4	12	20	50	2.5	4.9	3.4	3.5
Ex.5	12	15	10	2.5	9	-	-
Ex.6	12	15	20	2.5	8	-	-
Ex.7	12	15	30	2.5	7.2	-	-

\* in microns

\*\* as percent by volume of fusing surface layer

\*\*\* as parts per 100 parts by weight of Viton®A

\*\*\*\* relative cure level normalized to equivalent Cure 50 parts by weight in Composition 1.

- not measured

**[0249]** For the fuser rollers of Comparative Examples 1-4 and Examples 1-7, the equilibrium surface was reached at, or approximately at, the 200K count.

**[0250]** The gloss values for Comparative Examples 1 and 2 in Table 2 demonstrate that the presence of small fillers, at or below 5 um, produces a high gloss. Unfilled fluoroelastomer



also tends to generate a gloss that may be unacceptable for certain applications.

**[0251]** As can be seen by comparing the gloss values of Comparative Examples 3 and 4 with those of Examples 1 and 2, inclusion of the discontinuous phase domains lowers gloss level. In this regard, at their equilibrium surfaces, the Example 1 and Example 2 fuser rollers generated a gloss number less than or equal to 5. For Example 1, the initial higher gloss was due to the presence of the artifact remaining from fuser roller preparation; by the 200K point this artifact had been worn away, with the resulting equilibrium surface providing a gloss level as indicated.

**[0252]** The gloss values for Examples 3 and 5-7 demonstrate that increasing the proportion of the discontinuous phase lowers gloss level. As to Example 3 in particular, at its equilibrium surface the fuser roller generated a gloss value of less than 5.

**[0253]** Example 4 shows that the discontinuous phase domains, and the indenter particles of the concurrently filed application identified herein as Applicants' Docket Nos. 10167 and PAT00010, may be combined to generate images with a gloss number of significantly lower than 5.

**[0254]** With Comparative Examples 1-4, high gloss values were obtained even after the respective artifacts were worn away.

**[0255]** Finally, although the invention has been described with reference to particular means, materials, and embodiments, it should be noted that the invention is not limited to the particulars disclosed, and extends to all equivalents within the scope of the claims.